

EFFECTS OF VACUUM ON THE TENSILE PROPERTIES
OF MAGNESIUM SINGLE CRYSTALS

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ABSTRACT

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The tensile behavior of magnesium single crystals at a temperature of $26 \pm 2^\circ\text{C}$ was investigated at varying pressure levels from 760 to 8×10^{-9} torr. For crystals deformed at a constant linear strain rate of 0.061 min^{-1} , there was no apparent effect of the vacuum environment down to 10^{-6} torr. However, from a vacuum of 10^{-6} torr down to the lowest pressure attained (8×10^{-9} torr), the effect of the vacuum environment was to increase the strain at the end of the first linear (or easy glide) stage and to decrease the slope of this stage; the magnitude of this effect was found to be dependent on pressure. The slope of the second linear stage was also decreased at the lower pressure levels; however, this effect appears to be caused primarily by the vacuum effect on the first linear stage. The magnitude of the vacuum environment effect was also found to be dependent upon strain rate. The reduction in flow stress at 5×10^{-8} torr was observed to be about 10 pct for a strain rate of 0.0275 min^{-1} and 70 pct for a strain rate of 0.67 min^{-1} . No systematic effect of pressure on the critical resolved shear stress was observed. The data are interpreted in terms of a modified form of Mott's work hardening mechanism for hexagonal metals.

Author

N66-18333

GPO PRICE \$ _____

(ACCESSION NUMBER)
25

(THRU)

CFSTI PRICE(S) \$ _____

(PAGES)
TMX 56156
A CR OR TMX OR AD NUMBER

(CODE)

17
(CATEGORY)

Hard copy (HC) 1.00

Microfiche (MF) .50



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Dell P. Williams* and Howard G. Nelson*

It has long been known that surface conditions influence the mechanical behavior of materials. Roscoe¹ demonstrated the importance of surface conditions on mechanical behavior by testing cadmium crystals with oxide layers. He found that the "apparent" critical resolved shear stress for cadmium was increased approximately 2.5 times by the presence of an oxide layer 1000 atoms thick. The magnitude of this effect was dependent on film thickness with crystals having films less than 20 atoms thick still showing a definite increase. This observed strengthening could not be attributed to any strength of the oxide film because of (1) the thinness of the films, and (2) an observed increase in the flow stress as deformation proceeded. Harper and Cottrell² obtained similar results on zinc crystals which had been etched and steamed to give artificially thick oxide films. However, in the course of this study, they also carefully examined the very early stages of plastic flow for zinc crystals which, in one case, had been electrolytically polished, and, in the other, had been lightly etched to produce a thin film. They then found that the critical resolved shear stress for both surface treatments of the crystals was actually identical but that the etched crystal exhibited a much more rapid initial hardening rate. This had not been noted previously. This result indicated that, in fact, the film caused an increase in the stress for continuation of slip, rather than for the initiation of slip.

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Lipsett and King³ investigated the effect of evaporated gold films on the plastic behavior of cadmium single crystals. For large angles (≈ 75 deg) between the specimen axis and the normal to the slip plane, they found that the stress-strain curve of the coated specimen always lies above that for a clean specimen. The shear strain at the end of the first linear stage was greater in clean specimens than in coated specimens and the slope of the second linear stage was less in the clean specimens than in the coated specimens. Recent results by Kramer and Podlaseck⁴ on aluminum crystals in high vacuum indicate that even the thin oxide films normally found on polished metal surfaces can significantly influence mechanical behavior.

Independently, Mott^{5,6} postulated a model for work hardening which he discussed in terms of hexagonal metals. He suggested that the edge dislocations on the basal plane would be free to glide out of the crystal with relatively little interruption due to an almost complete absence of nonbasal slip with this crystal structure. Thus, the only barriers to edge dislocations would be oxide or other type films on the surface. The screw dislocations, however, would pile up against other screws which have acquired sufficient jogs to stop their movement. The jogged screw dislocations then would constitute a forest of dislocations which would hinder further dislocation motion. If some of the sources of dislocations on prismatic or pyramidal planes become active during the first linear stage, hardening then could be explained by an increase in the density of jogged dislocations and the consequent increase in the density of piled-up groups of screws. The transition from the first to the second linear stage would then correspond to a sudden increase in the activity of nonbasal slip systems. Mott has suggested that this sudden activity is due to the back stress caused by piled-up groups of screws.

The postulates of Mott's model might be consistent with the experimental results of references 1, 2, and 3, if the effect of surface barriers was considered to be

significant. It was therefore felt that there could be substantial changes in the strain hardening characteristics of a hexagonal metal when subjected to high vacuum. The purpose of the present investigation was to determine these effects, and, as possible, to relate them to the strain hardening mechanism.

EXPERIMENTAL PROCEDURES

Material and Specimen Preparation. The magnesium melting stock used in the growth of the single crystal specimens was triply sublimed magnesium which had been extruded to a 1/2-in.-diameter rod. The spectrographic analysis supplied with this stock indicated an impurity content less than the weight percentages shown: 0.005 Al, 0.01 Ca, 0.001 Cu, 0.0003 Fe, 0.001 Mn, 0.0004 Ni, 0.003 Pb, 0.001 Si, 0.01 Sn, and 0.006 Zn. Thus, the material exhibited a purity greater than 99.96 pct Mg.

Magnesium crystals were grown by directional solidification in a high-purity graphite mold under an argon atmosphere in a vertical furnace similar to that described by Jillson.⁷ Crystals, having orientations of the normal to the basal plane of 51, 44, and 15 deg and corresponding slip directions of 39, 54, and 75 deg, respectively, with deviations of ± 1 deg, were grown from seeds selected from a series of randomly grown seeds. The as-grown crystals were 1/2 in. in diameter and 6 in. long. Orientation was determined (to an accuracy of $\pm 1/2$ deg) and checked in each crystal by the Laue back-reflection technique in combination with Greninger's method of interpretation. Four photograms were taken at 90 deg intervals in a helical pattern along the length of each crystal. For all crystals tested, the lineage structure indicated by the photograms varied less than 1/2 deg.

The acceptable crystals were separated from the seed and cut into 3-in. lengths by the use of an acid cutting technique. A gage section with an active

length of 1-1/2 in. and a diameter of about 1/4 in. was acid machined in each crystal specimen by masking the ends and rotating the specimen at 25 rpm in an aqueous solution of 30 pct hydrochloric acid. No dimensional eccentricity could be detected in the gage length.

Several gripping techniques were investigated to find the most suitable method of gripping the crystals. It was found that machined threads were the most acceptable. For threading, the crystals were clamped in the lathe at a sufficient distance from the gage section such that no detectable distortion occurred in this section when checked with X-ray diffraction. Light cuts were made at all times to minimize bending of the crystal.

Crystal specimens were tested after having been given a 1-hr anneal at 400°C, a 1-hr anneal at 300°C, and no anneal. It was found that the shape of the tensile curves of all specimens given the two different anneals was nearly identical. All subsequent specimens tested were given a 1-hr anneal at 300°C to relieve any possible distortion due to handling. All tests were conducted within 48 hr after annealing. Several specimens were sectioned longitudinally through the machined grips following the anneal to determine the extent of growth of grains induced during the machining of threads. In every case growth was not sufficient to penetrate into the gage section of the crystal.

Before testing, the prepared crystals were polished by gently rolling in a tray containing 25 pct hydrochloric acid in water. After polishing they were successively rinsed in tap water and in acetone and allowed to dry in air. All tests were conducted within 24 hr after polishing.

Test Apparatus and Procedure. Tensile tests were conducted in a specially constructed vacuum chamber which was designed to fit between the columns of a Baldwin testing machine. Fig. 1 is a sketch of this system. Vacuum levels as

low as 5×10^{-9} torr were attained in the test chamber by the use of an ion-gettering pumping system. This system incorporated a sorption roughing pump, a 400 liter per sec ion pump, and a titanium evaporator sublimation pump. The cantilevered test chamber and pumping system were caster mounted so that the chamber could be removed from the testing machine for specimen replacement. The specimen was mounted in the test chamber by screwing the threaded ends into self-aligning pin grips. Tensile forces were applied to the specimen from the testing machine by the use of bellows-sealed connecting rods.

Tests were conducted at constant linear strain rates, $\dot{\epsilon}$, of 0.025, 0.061, 0.081, and 0.67 min^{-1} at a temperature of $26 \pm 2^\circ\text{C}$. Load and displacement were measured by an SR-4 type load cell having a 100-lb range and a 500-lb range and a deflectometer, respectively. Load was measured with a precision of 0.5 pct of full scale and strain with a precision of 6.67×10^{-3} mm per mm. The load-displacement curve was recorded during the test.

After polishing, the specimens were mounted in the vacuum chamber and connected by the bellows arrangements to the testing machine. The chamber was then evacuated to the desired pressure. Pressure was monitored continuously by a nude ion gage and periodically by a partial pressure analyzer. It was estimated that the total pressure values at the lower pressures ($< 10^{-7}$ torr) were subject to an error of nearly one-half a decade, but that the values at higher pressures ($> 10^{-7}$ torr) were more nearly correct. During a test, the total pressure varied slightly, but in all cases these variations were less than half a decade of pressure. Although detailed studies were not made, it was observed that, over the pressure range employed, the gas composition remained essentially the same as that of atmospheric air.

Each test was conducted at a constant linear strain rate. By varying both the load and strain magnification factors during the test, it was possible to

determine with precision the slopes and extents of the first and second linear hardening stages. In three cases the tests were interrupted at the reduced pressure and the stress was dropped to a value slightly below the initial yield stress. Air was then introduced to raise the pressure to atmospheric pressure. After waiting for temperature and pressure equilibration, the specimen was reloaded and the test continued.

The load-displacement curve obtained directly from the experimental apparatus for each test conducted was converted to a resolved shear stress versus shear strain by employing relations for this conversion developed by Schmid and Boas.⁸ The resolved shear strain for single crystals loaded in tension has been shown to be

$$\gamma = \frac{1}{\cos \varphi_0} \left(\sqrt{(1 + \epsilon)^2 - \sin^2 \lambda_0} - \cos \lambda_0 \right) \quad [1]$$

where γ is the resolved shear strain; φ_0 is the angle between the normal to the slip plane and the tensile axis at zero deformation; λ_0 is the angle between the slip direction and the tensile axis at zero deformation; and ϵ is the dimensionless elongation within the active gage length.

The resolved shear stress for single crystals under like conditions has been shown to be given by

$$\tau = \frac{L}{A} \cos \varphi_0 \cos \lambda_1 \quad [2]$$

where τ is the resolved shear stress; A is the cross-sectional area in the active gage section; L is the applied load; and λ_1 is the instantaneous value

of the angle between the slip direction and the tensile axis for a given elongation. The angle λ_1 is obtained from the equation

$$\sin \lambda_1 = \frac{\sin \lambda_0}{1 + \epsilon} \quad [3]$$

In employing the above relations, it was assumed that all macrodeformation occurred on the basal plane, $\{0001\}$, in the $\langle 11\bar{2}0 \rangle$ slip direction. This point was partially verified by metallographic examination of the specimen surface following deformation. In all cases, the $\{0001\} \langle 11\bar{2}0 \rangle$ slip system was the only operative slip system observed.

EXPERIMENTAL RESULTS

Effect of Pressure. Stress-strain curves were obtained in tension under room temperature conditions, $26 \pm 2^\circ\text{C}$, at 760 torr and at vacuum levels down to 8×10^{-9} torr. This series of tests was conducted at a constant linear strain rate of 0.061 min^{-1} .

Fig. 2 shows the effect of varying vacuum level on the tensile properties of magnesium single crystals having $\phi_0 = 44$ deg and $\lambda_0 = 54$ deg. For reference, the stereographic triangle and arrows indicating the end of the first and the start of the second linear stages have been included. As can be seen in the figure, a reduced pressure generally lowered the curves below those observed in air. Some variation of results is observed at high vacuum; this is believed to be due, primarily, to the random error inherent in the measurement of vacuum levels.

Fig. 3 shows the effect of varying vacuum level on the tensile properties of magnesium single crystals having $\phi_0 = 15$ deg and $\lambda_0 = 75$ deg. There is the same general effect of reduced pressure on the stress-strain curves of these crystals as that exhibited by crystals in Fig. 2 ($\phi_0 = 44$ deg and $\lambda_0 = 54$ deg).

Fig. 4 shows the salient features of the first linear stages of Fig. 2. This figure shows as a function of pressure the pct change, from that observed under atmospheric conditions, in strain at the end of the first linear stage, γ_1 ; in slope of the first linear stage, θ_1 ; and in stress at the end of this stage, τ_1 . It is seen that γ_1 and θ_1 are affected by the reduced pressure. Only a minor increase in γ_1 was exhibited at pressures in the range of 10^{-6} torr, with increases up to 80 pct observed in specimens tested at about 10^{-8} torr. The effect of reduced pressure on θ_1 has the opposite trend to that for γ_1 with a decrease in slope of about 70 pct observed at a pressure of about 10^{-8} torr. Because γ_1 and θ_1 are nearly mirror images of one another, τ_1 is found to be essentially independent of pressure variation. Similar comparisons could not be made for the results of Fig. 3 since the first linear stage was not well defined.

Fig. 5 shows the salient features of the second linear stages of Figs. 2 and 3. This figure shows as a function of pressure the pct change, from that observed under atmospheric conditions, in strain at the beginning of the second linear stage, γ_2 ; in slope of the second linear stage, θ_2 ; and in stress at the beginning of this stage, τ_2 . It is seen that γ_2 and θ_2 vary with pressure for crystals of both orientations. However, crystals with $\phi_0 = 15$ deg and $\lambda_0 = 75$ deg exhibited a greater pressure effect in this second stage than did those with $\phi_0 = 44$ deg and $\lambda_0 = 54$ deg. This orientation dependence can be explained on the basis of a strain-rate effect to be discussed later. Although the linear strain rates for both orientations were identical, the shear strain rate of crystals having their normal to the basal plane (ϕ_0) at 15 deg was much greater than those having ϕ_0 equal to 44 deg. Next, it is seen from the figure that the three tests conducted in ambient atmosphere, after straining in vacuum near or into the second linear stage (see Fig. 2), exhibited similar reductions in θ_2

to those that were continued in vacuum. This result tends to indicate that the observed pressure effect on θ_2 is due to the testing history of the crystal. It is also seen from the figure that τ_2 appears to be independent of pressure within data scatter.

Effect of Strain Rate. Stress-strain tests were conducted at various constant linear strain rates under room temperature conditions in the presence of ambient atmosphere and at vacuum levels of approximately 5×10^{-8} torr. These constant linear strain rates, $\dot{\epsilon}$, were 0.025, 0.081, and 0.670 min^{-1} . Single crystal specimens used in these tests had $\phi_0 = 51$ deg and $\lambda_0 = 39$ deg.

Fig. 6 shows the effect of strain rate on the observed dependence of the flow stress on pressure. Tests conducted at atmospheric pressure and at strain rates of 0.025 and 0.67 min^{-1} indicate the normally expected strengthening which occurs with increasing strain rate. The magnitude of this strengthening is, of course, dependent on orientation, temperature level, and prior history. Variable strain-rate tests conducted at a vacuum level of 5×10^{-8} torr, however, show a trend opposite to that observed in air. In this case, an increase by a factor of 20 in strain rate decreased the flow stress by a factor of 2.

DISCUSSION

The experimental test results show that for magnesium single crystals strained at a constant linear strain rate, the effect of high vacuum is to lower the stress required for a given strain, as shown in Figs. 2 and 3. The stress-strain relation is essentially independent of pressure down to 10^{-6} torr; however, between 10^{-6} and the lowest test pressures, the reduction is dependent on pressure. In this section, the observed results will be discussed in terms of a proposed qualitative model for work hardening of hexagonal metals. The various points in the model will be compared to a mechanism suggested by Mott.^{5,6}

The crystals used in this investigation all had a surface layer of normal oxide film. This oxide is quite stable and should neither sublime nor decompose under high vacuum conditions at the test temperature (26°C). Thus, for the environmental conditions of this investigation, the crystals should exhibit the same critical resolved shear stress. This is consistent with the results of this investigation (Figs. 2 and 3).

From this investigation it is suggested that, at the yield point, some dislocations leave the crystal, causing a reduction in the average pileup length and a rupture of the contaminated surface layer. It is further suggested that, at strains greater than the yield strain, the average length of the pileup and the back stress created by it would be related to the rate at which dislocations arrive at the surface and to the rate of contamination of the newly exposed surface layer. Evidence of this is given by the variable strain-rate tests (Fig. 6) in which increasing strain rate at vacuum pressures of 5×10^{-8} had the same effect on the curves as would be expected from further reductions in pressure. As deformation continues, more pileups would be created at both the internal and surface barriers. The strength and density of these barriers would then determine the flow stress for a given strain. The strength and density of the internal barriers are dependent on such well-known factors as orientation, strain rate, temperature, etc.; thus, they should be relatively independent of environment. The average strength and density of the surface barriers, however, are directly dependent on environment.

It is suggested that the end of the first stage is determined by a value of the back stress at which a different deformation mechanism becomes operative. Fig. 4(c) shows that the resolved shear stress at the end of the first stage is essentially constant, regardless of vacuum level. Thus, the value of the back

stress responsible for activation of a secondary mechanism must be due not only to the stress contribution of internal barriers but also to the contribution of surface barriers and the resulting pileups.

Since the mechanism that has generally been proposed for hardening of the second linear stage is based on nonbasal slip and a rapid increase in internal barrier formation, one might expect this portion of the flow curve to be independent of environment. The results of this investigation indicate that the slope of the second linear stage has a similar environmental trend as that of the first stage. However, this dependence apparently originates from the environmental history of the first stage, as shown by tests interrupted and continued in air, Fig. 5. The experimental results are at present too few for a detailed discussion of this observation; however, a possible explanation may lie in the increased rate of internal barrier formation in the second linear stage due to orientation and specimen configuration considerations.

The model which has been proposed is similar to that suggested by Mott,^{5,6} but with some important differences. As noted previously, Mott has suggested that the very long first stage exhibited by hexagonal metals is characteristic of long slip distances and may be explained by large numbers of edge dislocations gliding out of the crystal. He feels that the slight hardening observed in this region is due to the hindrance to motion of some dislocations by jogged screw dislocations threading the basal plane. These jogged screws would be the result of small amounts of nonbasal slip. The results of the present investigation suggest that although some edge dislocations do glide out of the crystal, the contaminated surface layer acts as a barrier and exerts a back stress which must be overcome by dislocations exiting from the crystal. Thus, the hardening behavior is a result not only of the jogged screws and the pileups resulting therefrom, but also of the surface barriers and the piled-up edge dislocations.

Mott has suggested that the end of the first linear stage is determined by a value of the back stress at which appreciable nonbasal slip commences. This is consistent with the results of this investigation so long as it is noted that the critical value of back stress must be due to both surface and internal barriers.

The mechanism by which a high vacuum environment can affect the mechanical behavior of a metal has been under study by various researchers for many years. Without attempting to review all the proposed theories, most work is based on the effects of the vacuum environment on the creep and fatigue behavior of polycrystalline metals. Kramer and Podlaseck⁴ have suggested that as the partial pressure of oxygen is reduced, the rate of formation of an oxide film on the freshly exposed slip steps is decreased and the time interval for dislocations to flow without obstruction through the surface is increased. This appears to be a likely explanation of the current results. At relatively high vacuum levels ($> 10^{-6}$ torr), a complete and normal oxide film would be produced and the behavior would be pressure independent. At moderately low vacuum levels (10^{-6} to 10^{-9} torr), the mechanism would suggest that the average density and length of surface pileups, and consequently the back stress, would be a function of oxygen partial pressure. At some lower pressure the rate of oxide formation would be negligibly small compared to the rate at which new surface is created and the results would again be pressure independent. The pressure range over which this would occur would be dependent on the thermodynamics and kinetics of oxide formation, as well as the strain rate employed. As can be noted from Fig. 6, the results agree well with this hypothesis since they show the magnitude of the environmental effect at a constant pressure to be strongly strain-rate dependent.

SUMMARY

The effect of pressures from 760 to 8×10^{-9} torr on the tensile behavior of magnesium single crystals was investigated. Based on the results obtained and the foregoing discussion, the following remarks can be made:

1) For magnesium single crystals strained at a constant linear strain rate of 0.061 min^{-1} , the stress required for a given strain is independent of vacuum level from 760 to 10^{-6} torr. At vacuum levels from 10^{-6} to 10^{-8} torr (the lowest pressure attained) the stress required for a given strain decreases with decreasing pressure and the reduction is more pronounced at the higher strain rates.

2) The effect of reduced pressure on the stress-strain curve appears to be related to the mechanisms associated with the first linear stage of hardening. The effect of high vacuum is to increase the strain at the end of this stage and to decrease the slope. There is no apparent effect on the stress at the end of the first linear stage. The apparent effect upon the second linear stage appears to depend upon the behavior of the first linear stage.

3) The data indicate that the deformation behavior of magnesium single crystals during first stage hardening can be explained in terms of Mott's work hardening mechanism as applied to hexagonal metals, if the effects of surface barriers are included.

This investigation is continuing to more clearly define the mechanism by which the high vacuum environment effects the hardening of hexagonal metals. This work will be concerned with the surface chemistry involved in oxide formation and further mechanical behavior of both polycrystalline and monocrystalline hexagonal metals, particularly at higher vacuums than presently attainable.

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FIGURE CAPTIONS

- Fig. 1.- Schematic drawing of vacuum system and test apparatus.
- Fig. 2.- The effect of vacuum on the tensile stress-strain curve for single crystal magnesium, $\phi_0 = 44$ deg and $\lambda_0 = 54$ deg.
- Fig. 3.- The effect of vacuum on the tensile stress-strain curve for single crystal magnesium, $\phi_0 = 15$ deg and $\lambda_0 = 75$ deg.
- Fig. 4.- The effect of vacuum on the slope and extent of the first linear stage for magnesium single crystals.
- Fig. 5.- The effect of vacuum on the slope and initiation of the second linear stage for magnesium single crystals.
- Fig. 6.- The effect of strain rate on the tensile stress-strain curve in air atmosphere and vacuum ($\sim 5 \times 10^{-8}$ torr) for single crystal magnesium, $\phi_0 = 51$ deg and $\lambda_0 = 39$ deg.

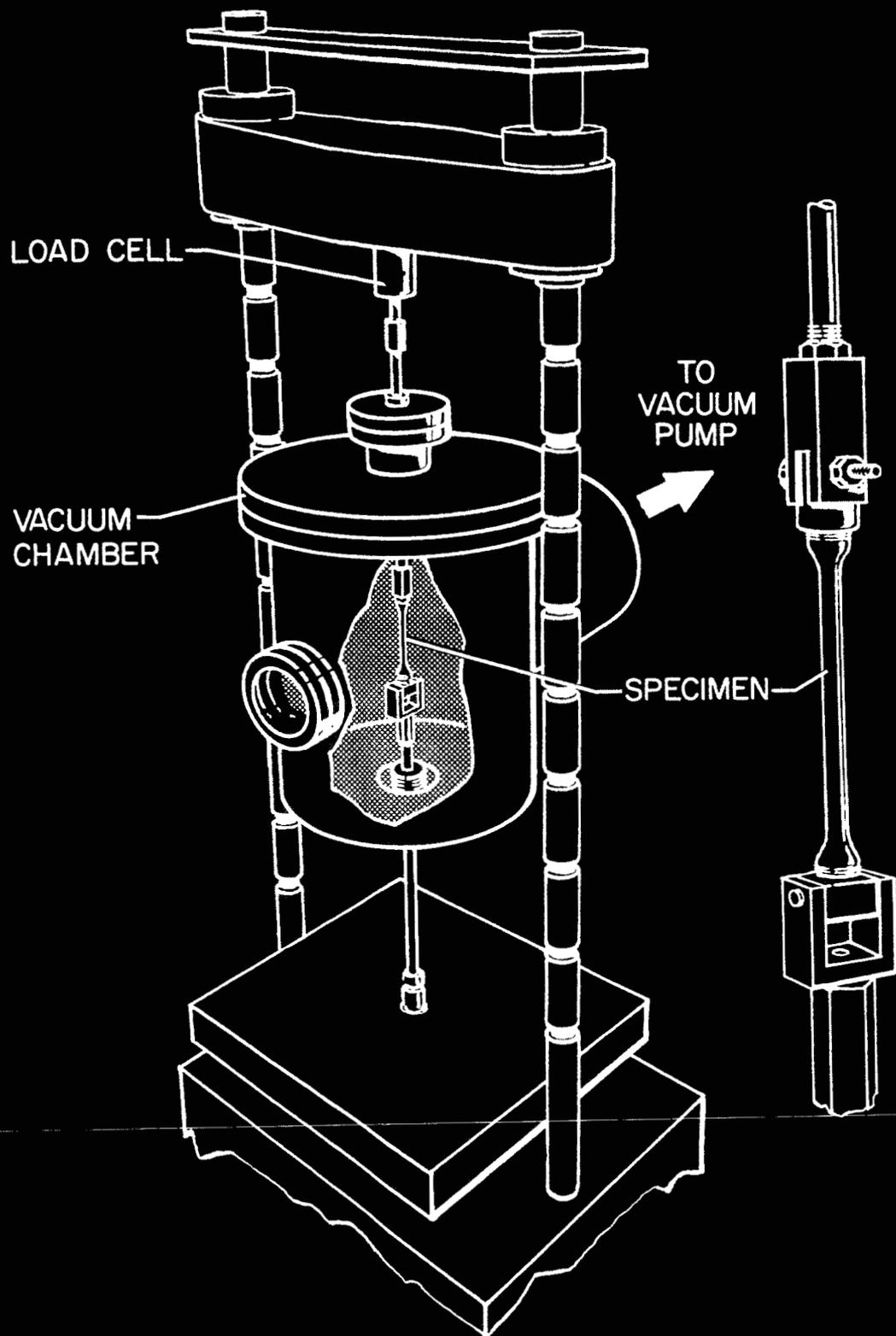


Fig. 1.- Schematic drawing of vacuum system and test apparatus.

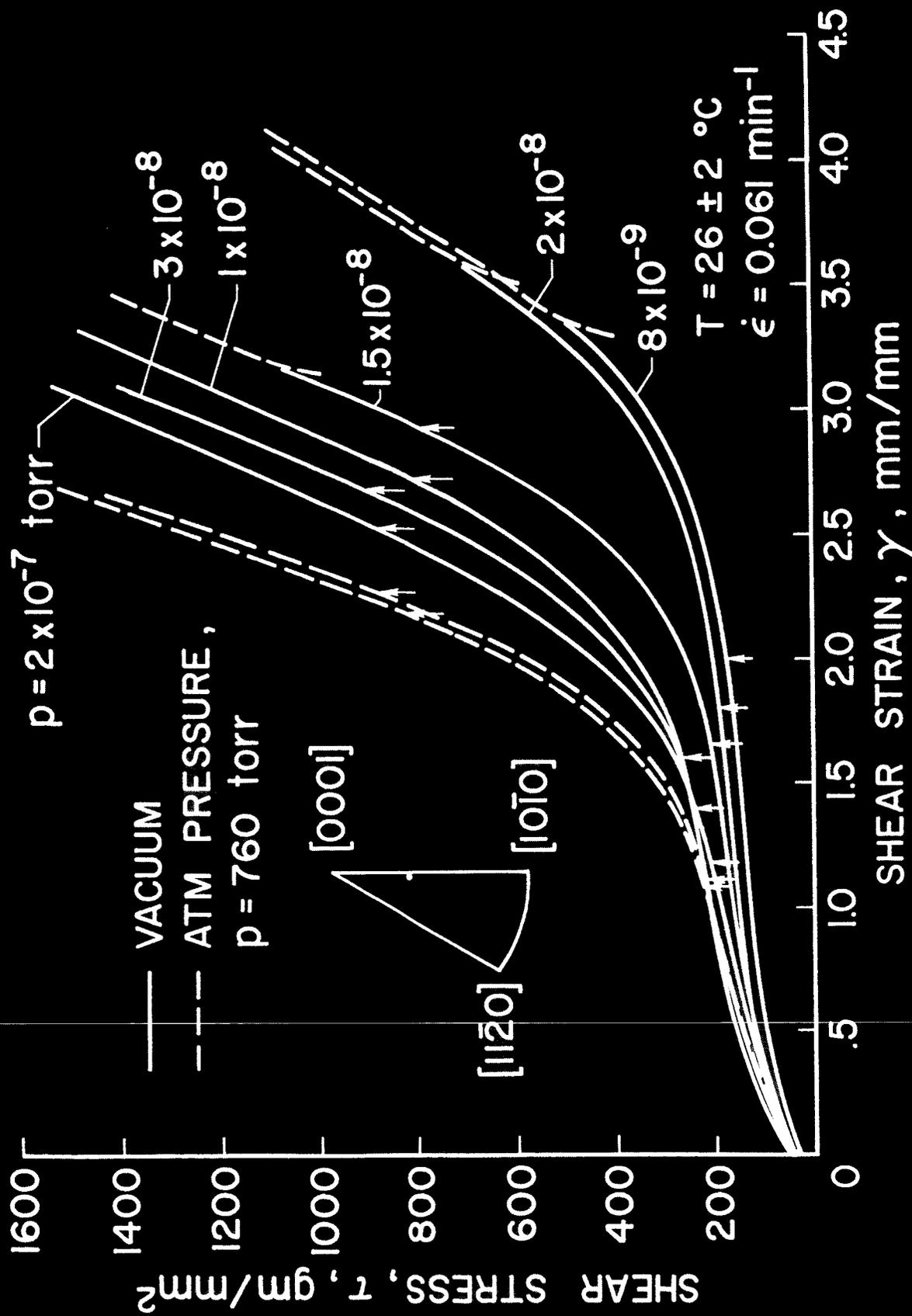


Fig. 2.- The effect of vacuum on the tensile stress-strain curve for single crystal magnesium, $\phi_0 = 44$ deg and $\lambda_0 = 54$ deg.

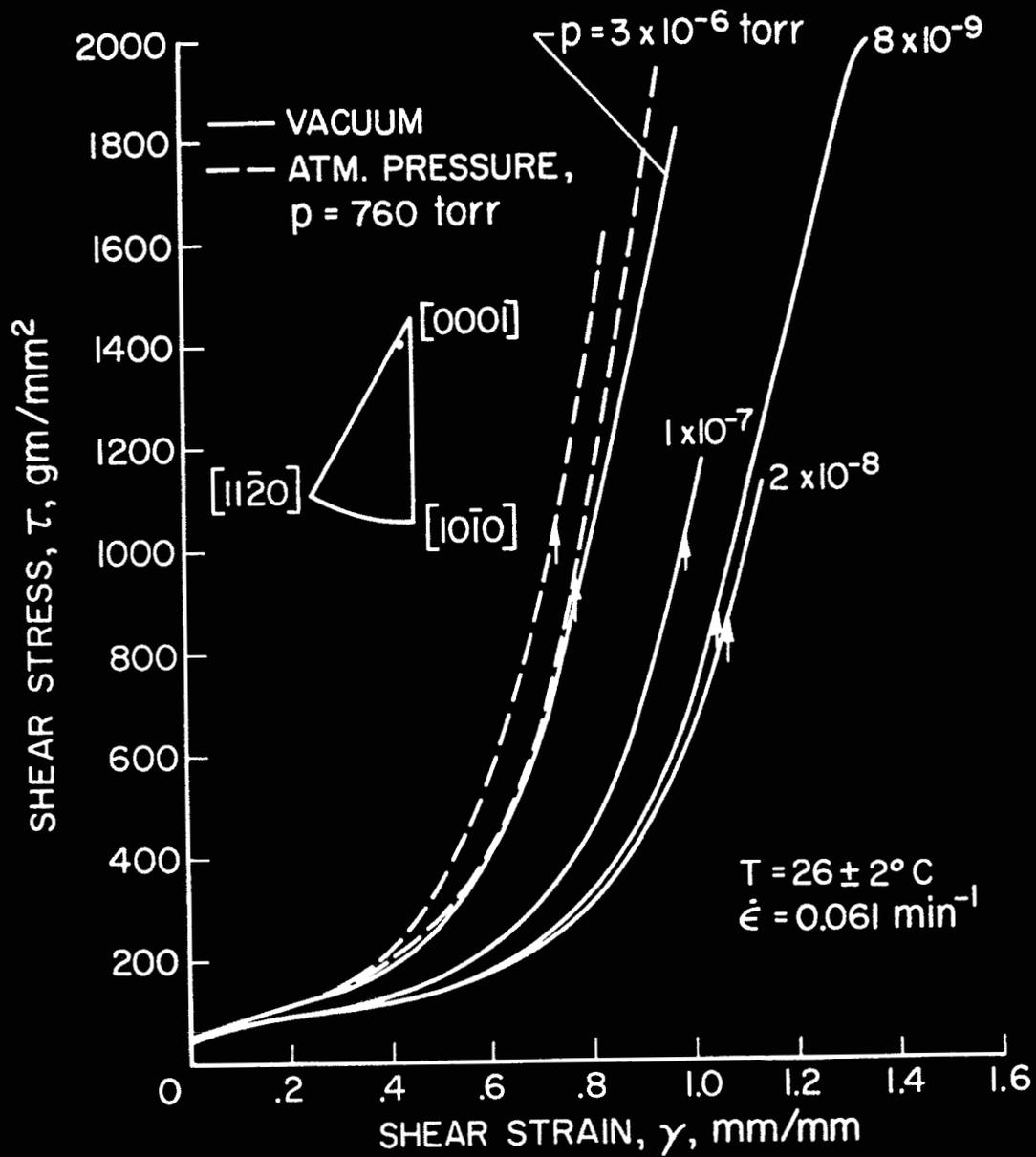


Fig. 3.- The effect of vacuum on the tensile stress-strain curve for single crystal magnesium, $\phi_0 = 15$ deg and $\lambda_0 = 75$ deg.

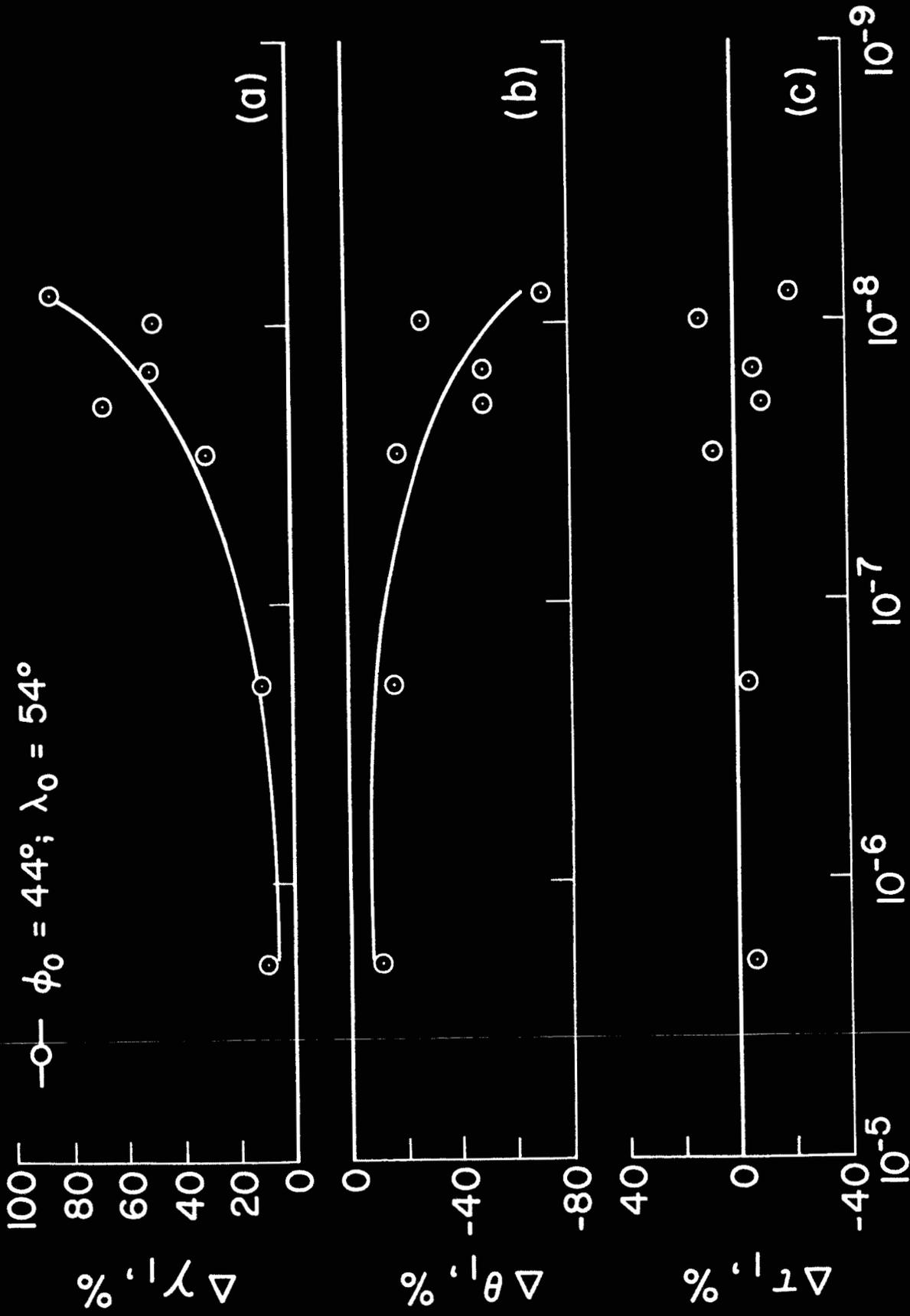


Fig. 4.- The effect of vacuum on the slope and extent of the first linear stage for magnesium single crystals.

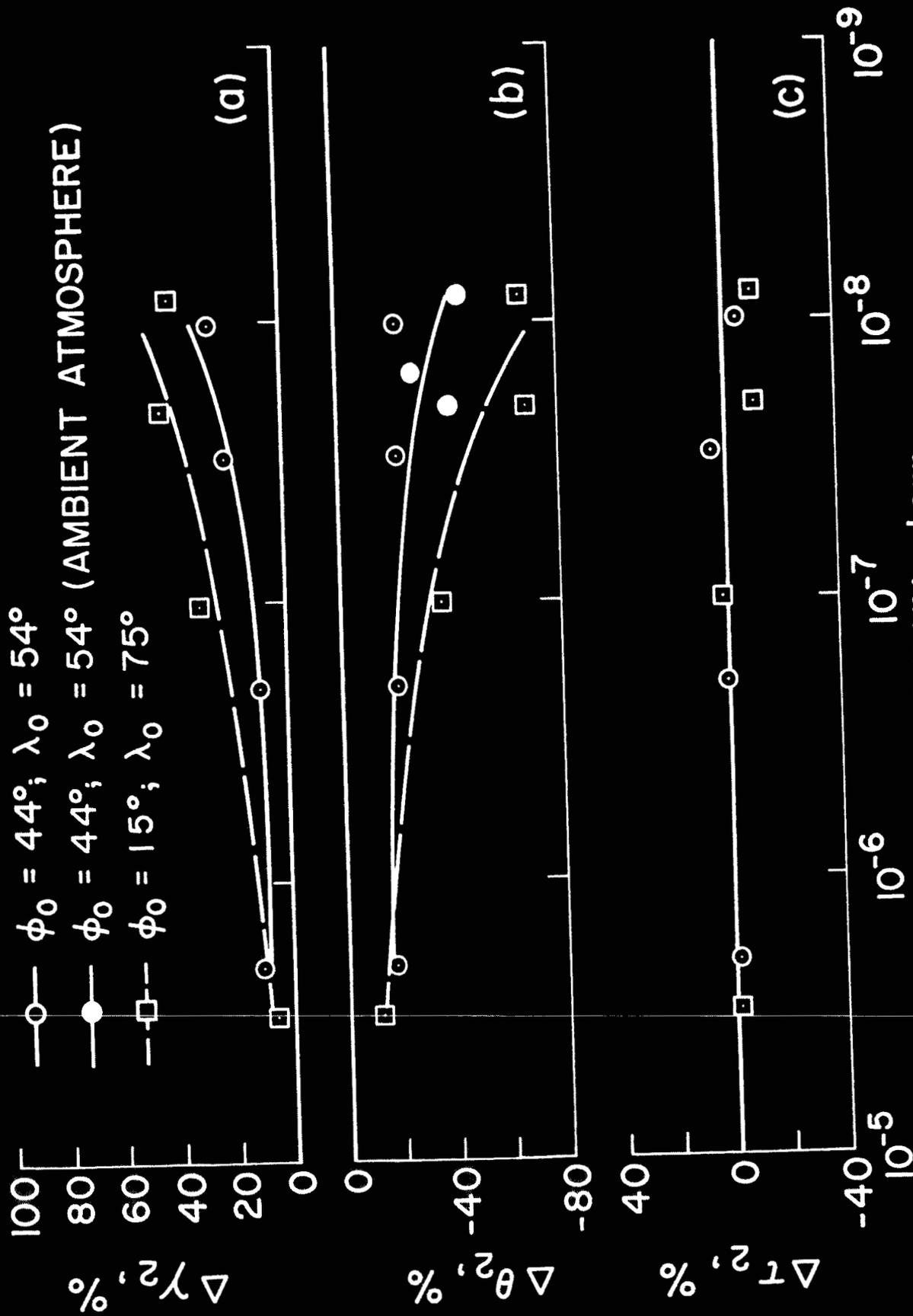


Fig. 5.- The effect of vacuum on the slope and initiation of the second linear stage for magnesium single crystals.

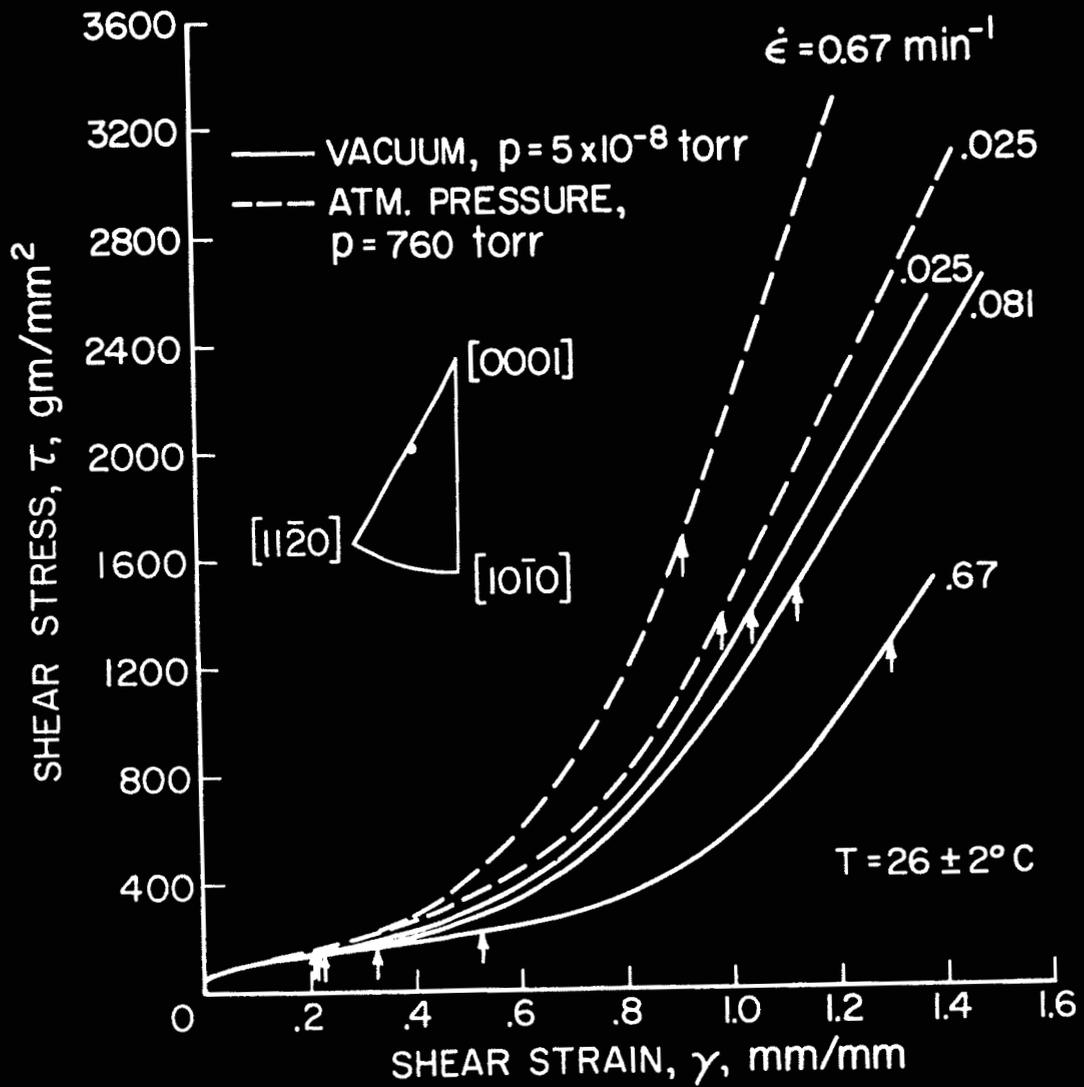


Fig. 6.- The effect of strain rate on the tensile stress-strain curve in air atmosphere and vacuum ($\sim 5 \times 10^{-8}$ torr) for single crystal magnesium, $\phi_0 = 51$ deg and $\lambda_0 = 39$ deg.